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SYNTHESIS OF FERROCENE-NITROGEN-CONTAINING POLYMERS WITH CONJUGATED BONDS

by
T. P. Vishnyakova
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Ya. M. Paushkin

Vysokomolekulyarnyye Soyedineniya, 8, No. 1, 181-185 (1966).

Translated from the Russian by Robert C. Taylor

October 1966

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Translation Branch
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U. S. Army Missile Command
Redstone Arsenal, Alabama 35809

In connection with the stormy development of new branches of technology the necessity has recently arisen for the creation of polymers which possess a number of specific properties: high thermal stability, increased electrical conductivity, and catalytic and magnetic properties. In connection with this, polymers which contain a system of conjugated bonds and also heteroatoms (nitrogen atoms, for example) and metallocycles in the conjugation chains are of great interest.

Starting with various nitriles in the presence of $ZnCl_2$, Kargin and his co-workers[1,2] obtained polymers with a system of C = N-bonds. Other authors[3,4,5] obtained polymer compounds with alternated C = N-bonds by the condensation polymerization of carbamide, ammonium carbonate, and ammonium bicarbonate and by the heterocondensation polymerization of acetaldehyde with ammonium bicarbonate.

The synthesis of only two ferrocene-nitrogen-containing polymers with conjugated double bonds is described in the literature: namely, polyazines, which are obtained by the condensation polymerization of 1.1'-diethylferrocene with hydrazine[6] and polyasephenyleneferrocenes, which are obtained by the reaction of 4.4'-bisdiazobiphenyl and 3.3'-dicarboxylic acid-4.4'-bisdiazobiphenyl with ferrocene[7].

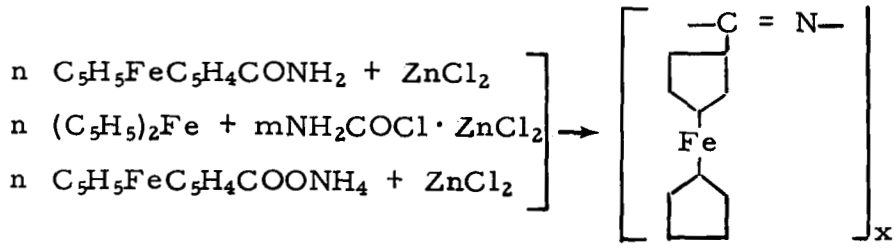
The authors obtained new ferrocene-nitrogen-containing polymers with a system of conjugated bonds—polyferrocenenitriles—on the basis of amides and ammonium salts of ferrocenecarboxylic acids.

1-ferrocenecarboxylic acid and 1.1'-ferrocenedicarboxylic acids were obtained by the oxidation of acetyl- and -1.1'-diacetylferrocene with potassium hypochlorite[8] with a corresponding yield of 42% and 92% of the theoretical yield. Their ammonium salts were obtained by passing ammonia gas through ferrocenecarboxylic acids in a dimethylformamide solution. Diamides of 1.1'-ferrocenedicarboxylic acid (dicarbamylferrocene) were obtained with an almost quantitative yield by passing ammonia gas through a benzol solution of 1.1'-ferrocenedicarbonylchloride which was obtained during the reaction of 1.1'-ferrocenedicarboxylic acid with phosphorus trichloride (the yield was 46% of the theoretical yield). The carbamylferrocene was synthesized from ferrocene and a $NH_2COCl \cdot AlCl_3$ complex by the method proposed by Little and Eisenthal[9].

The condensation polymerization was conducted in a test-type autoclave in the absence of the oxygen of the air. Zinc chloride was used as a catalyzer. Upon termination of the reaction the unreacted monomers were washed out of the products and their solubility was then checked in various organic solvents. The polymers were partially

dissolved in dimethylformamide from which the water had been precipitated. The undissolved fraction was rinsed with a 10% solution of hydrochloric acid until a negative reaction on the zinc and iron ions and with distilled water until a negative reaction on the Cl^- ions was obtained. The polymers thus obtained were dried in a vacuum at 40°-50°C to a permanent weight.

Polyferrocenylnitrile was obtained by the condensation polymerization of carbamylferrocene, of ammonium salts of ferrocenecarboxylic acid, and also directly from ferrocene and carbamylchloride with a zinc chloride complex:



P_2O_5 and $TiCl_4$, besides zinc chloride, were also used as catalyzers during the condensation polymerization of carbamylferrocene.

The data which characterizes the activity of these catalyzers are shown in Table I.

Table I. A Comparative Evaluation of the Activity of the Catalyzers Which Were Employed During the Condensation Polymerization of Carbamylferrocene

(140°C, 5 hours, monomer: catalyzer ratio—1 : 1)

Catalyzer	Polymer Yield (% of Theoretical Yield)			Elementary Composition (%)*			
	Dissolved in Dimethylformamide	Undissolved	Overall	C	H	N	Fe
$ZnCl_2$	14.4	46.0	60.4	63.65	4.41	6.89	25.97
$TiCl_4$	18.1	13.3	31.4	62.9	4.4	6.91	26.05
P_2O_5	Het	14.5	14.5	62.7	4.38	6.54	25.7

*$C_{11}H_9FeN$. Calculated (yield), %: C, 62.6; H, 4.27; N, 6.63; Fe, 26.5.

While obtaining polyferrocenylnitrile by various methods the effect of the reaction conditions (temperature, duration, monomer-catalyzer ratio) on the polymer yield was studied. The experiments were conducted at temperatures of from 80° to 350°C and with a reaction duration of from 1.5 to 8 hours.

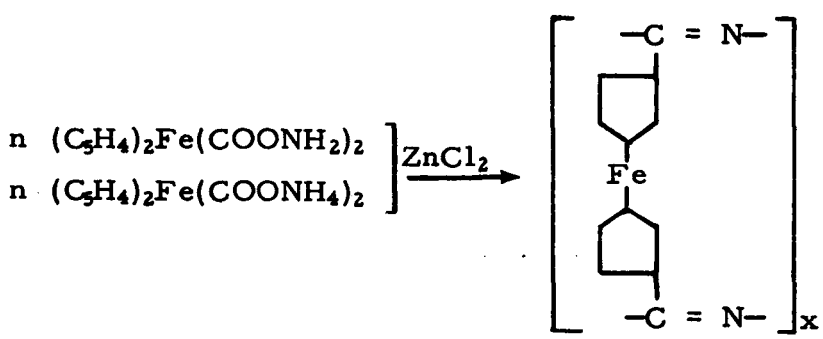
A comparative evaluation of the three methods of polyferrocenylnitrile synthesis is given in Table II. As is evident from the table, the simplest and most effective method is its synthesis from ferrocene and a carbamylchloride with zinc chloride complex. The polymer yield during this method was 87.0% of the initial ferrocene.

Table II. An Evaluation of Methods of Polyferrocenylnitrile Synthesis

Initial Reagents	Reaction Conditions*	Polymer Yield (%)		Elementary Composition, (%)***			
		From the Theoretical Yield	Based on the Initial Ferrocene (Added)	C	H	N	Fe
Carbamylferrocene + $ZnCl_2$	170°, 5 hours M : K - 1 : 1	67.7	47.8	63.56	4.41	6.89	25.97
Ferrocene + $NH_2COCl \cdot ZnCl_2$	180°, 5 hours**	87.0	87.0	61.9	4.52	5.67	26.56
Ammonium Salt of Ferrocenecarboxylic Acid + $ZnCl_2$	200°, 5 hours, M : K - 1 : 2	63.8	31.9	63.1	4.36	5.7	25.9

* M - monomer, K - catalyzer.
** Ferrocene: NH_2COCl : $ZnCl_2$ = 2 : 1 : 1.
*** $C_{11}H_9FeN$. Calculated (yield), %: C, 62.6; H, 4.27; N, 6.63; Fe, 26.5.

Polyferrocenyldinitrile was obtained by the condensation polymerization of diamide and diammonium salts of 1.1'-ferrocenedicarboxylic acid:



As is evident from Table III, the most convenient method is the synthesis of a polymer on the basis of diammonium salt of 1.1'-ferrocenedicarboxylic acid.

Table III. An Evaluation of Methods of Polyferrocenyldinitrile Synthesis

(200°C, 5 hours, monomer : catalyzer ratio - 1 : 2)

Initial Monomers	Polymer Yield (%)		Elementary Composition (%)*			
	From the Theoretical Yield	Based on Ferrocene	C	H	N	Fe
$(C_5H_4)_2Fe(CONH_2)_2$	52.3	22.2	60.34	3.38	9.38	21.9
$(C_5H_4)_2Fe(COONH_4)_2$	48.4	44.5	60.6	3.14	9.31	22.9

* $C_{12}H_8N_2Fe$. Calculated (yield), %: C, 61.0; H, 3.4; N, 11.9; Fe, 23.7.

During polyferrocenyldinitrile synthesis by two methods the effect of the various reaction conditions on the polymer yield was also studied; the optimum conditions of synthesis and the polymer yields are shown in Table III.

The products of the condensation polymerization were black to brown colored powders, depending on the conditions of the reaction. All of the undissolved polymers did not melt up to 500°C, and the dissolved polymers melted in the temperature range of from 350° to 400°C. The elementary composition of the products obtained, as is evident from Tables II and III, corresponds with the proposed structure.

The infrared spectra taken for the obtained polymers also confirm the proposed structure (Figure 1). In the spectra of all the polymers an 820 cm^{-1} band of absorption was found, which is characteristic for ferrocene compounds. For polyferrocenylnitrile, bands of absorption are found in the region of 1000-1100 cm^{-1}, which is characteristic for a free ferrocene cyclopentadiene ring; which tells us that condensation polymerization proceeds along one cyclopentadiene ring.

In the spectra of all the polymers there is also intensive absorption in the region of 1600 cm^{-1} which is apparently caused by the valence oscillations of the C = N-bonds.

The properties of the synthesized polymers are set forth in Table IV.

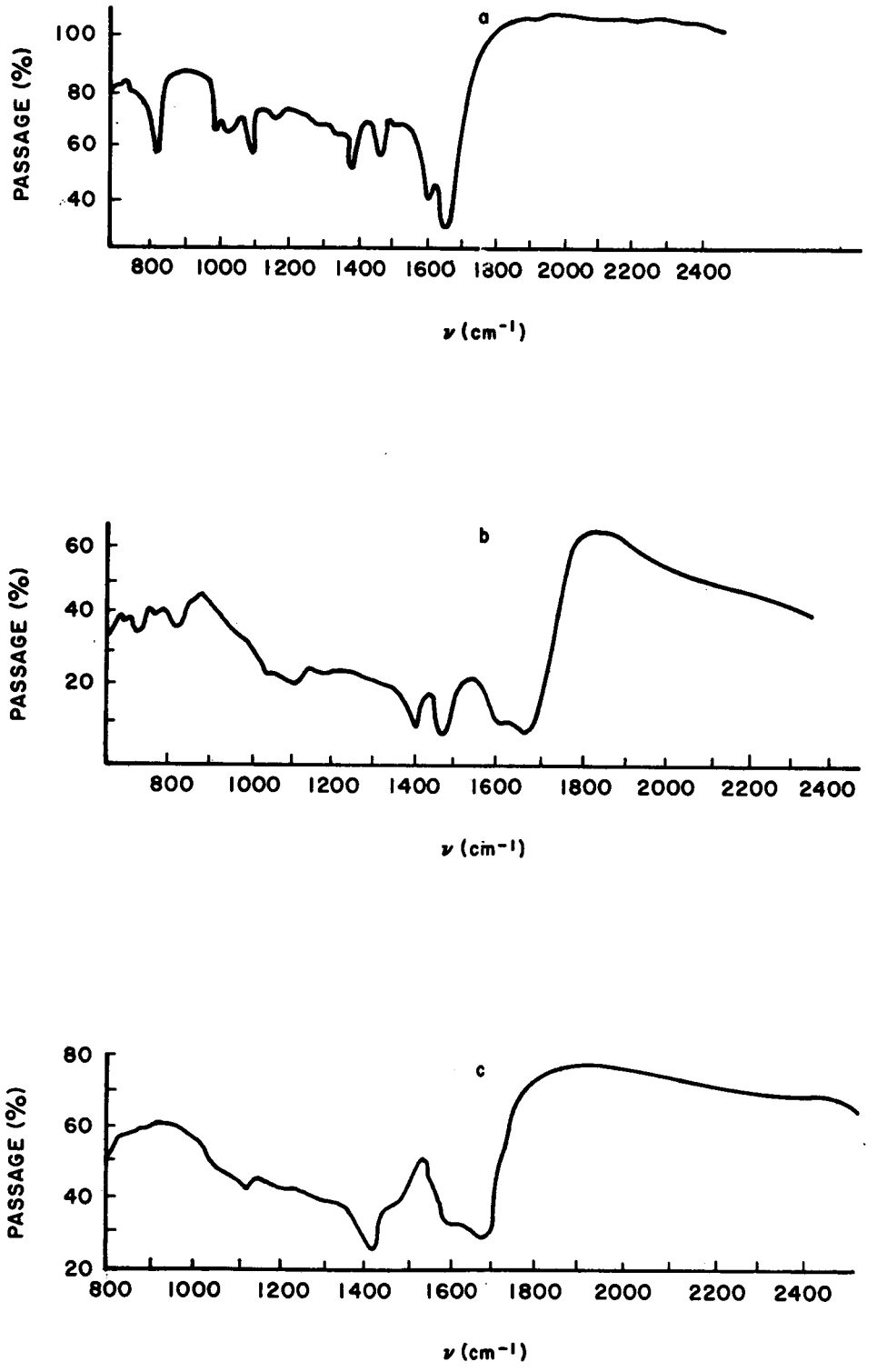
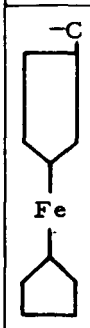
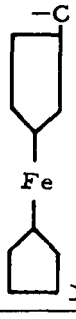


Figure 1. The Infrared Spectrum of Polyferrocenylnitriles Obtained from: a - Carbamylferrocene; b - Ferrocene and $NH_2COCl \cdot ZnCl_2$; c - Dicarbamylferrocene

Table IV. The Properties of Polyferrocenylnitriles

Polymer Structure	Melting Point (°C)		Molecular Weight	N (spin/g)	σ_{50} ($ohm^{-1} \cdot cm^{-1}$)	ΔE (ev)
	Dissolved	Undissolved				
	350-450	>500	1200-1600	10^{17}-10^{19}	10^{-11}-10^{-8}	0.724-0.09
	Het	>500	-	10^{18}	10^{-12}-10^{-14}	0.93-1.28

For soluble fractions of polymers the molecular weight, which lies within limits of from 1600 to 1200, was determined by the isopiestic method in dimethylformamide.

All polymers give a narrow single-component signal in the EPR spectrum which is characteristic for polyconjugated systems with a number of 10^{17}-10^{20} unpaired electrons per gram.

The electro-physical properties of polymers were studied. The dependence of the conductivity on the temperature is exponential, which is characteristic for semiconductors. The specimens investigated have a specific electrical conductivity of 10^{-8}-10^{-14} $ohm^{-1} \cdot cm^{-1}$ (Figure 2) at 50°C.

Conclusions

1. Polyferrocenylnitrile was obtained by the condensation polymerization of amide and the ammonium salts of ferrocenecarboxylic acid, and also directly from ferrocene and a carbamylchloride with zinc chloride complex.

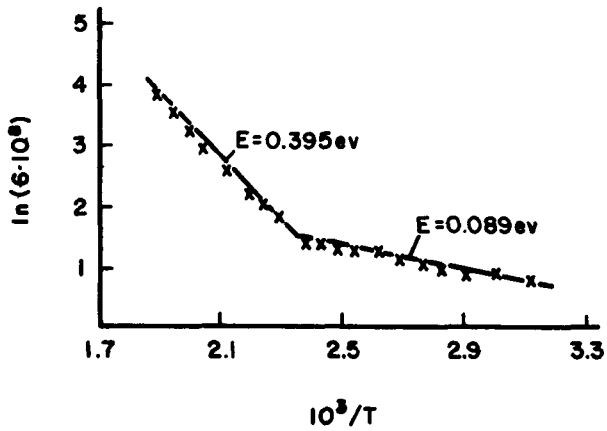


Figure 2. The Dependence of the Electrical Conductivity on the Temperature for Polymers Obtained from Ferrocene and $NH_2COCl \cdot ZnCl_2$

2. Polyferrocenyldinitrile was obtained by the condensation polymerization of diamide and the diammonium salts of 1.1'-ferrocenebicarboxylic acid.

3. The effect of various reaction conditions on the polyferrocenylnitrile yield was studied.

4. Polyferrocenylnitriles give a signal in the EPR spectrum with a number of 10^{17} - 10^{20} unpaired electrons per gram. The dependence of the electrical conductivity on the temperature bears an exponential nature.

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